

## **CERTIFICATION REPORT**

**Certification of Charpy V-notch reference test pieces of  
30 J nominal absorbed energy**

**Certified Reference Material ERM<sup>®</sup>-FA013bf**

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30 J nominal absorbed energy**

**Certified Reference Material ERM<sup>®</sup>-FA013bf**

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## Abstract

This certification report describes the processing and characterisation of ERM<sup>®</sup>-FA013bf, a batch of Charpy V-notch certified reference test pieces. Sets of five of these test pieces are used for the verification of pendulum impact test machines according to EN 10045-2 (Charpy impact test on metallic materials, Part 2. Method for the verification of impact testing machines [1]) or according to ISO 148-2 (Metallic materials - Charpy pendulum impact test – Part 2: Verification of test machines [2]).

The certified value for  $KV$  (= energy required to break a V-notched test piece using a pendulum impact test machine) is 27.7 J. The associated uncertainty (1.1 J,  $k = 2$  corresponding to a confidence level of about 95 %) is calculated for the mean of a set of five test pieces.

The absorbed energy ( $KV$ ) is procedurally defined and refers to the impact energy required to break a V-notched bar of standardised dimensions, as defined in EN 10045-1 [3] and ISO 148-1 [4]. The certified value of ERM<sup>®</sup>-FA013bf is traceable to the SI, via the SI-traceable certified value of the master batch ERM<sup>®</sup>-FA013ba, by testing samples of ERM<sup>®</sup>-FA013ba and ERM<sup>®</sup>-FA013bf under repeatability conditions on an impact pendulum verified and calibrated with SI-traceably calibrated tools. The certified value is valid only for strikers with a 2 mm tip radius. The certified value is valid at  $(20 \pm 2) ^\circ\text{C}$ .

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## Glossary

AISI	American Iron and Steel Institute
ASTM	American Society for Testing and Materials
BCR	Community Bureau of Reference
CEN	European Committee for Standardization
CRM	Certified Reference Material
EC	European Commission
EN	European Norm
ERM <sup>®</sup>	European Reference Material
h	Hour
IMB	International Master Batch
IRMM	Institute for Reference Materials and Measurements
ISO	International Organisation for Standardization
JRC	Joint Research Centre
$k$	Coverage factor
$KV$	Absorbed energy = energy required to break a V-notched test piece of defined shape and dimensions when tested with a pendulum impact testing machine
$KV_{CRM}$	Certified $KV$ value of a set of 5 reference test pieces from the Secondary Batch
$KV_{MB}$	Certified $KV$ value of the Master Batch test pieces
LNE	Laboratoire National de Métrologie et d'Essais
MB	Master Batch
min	Minute
$n_{MB}$	Number of samples of the Master Batch tested during certification of the Secondary Batch
$n_{SB}$	Number of samples of the Secondary Batch tested for certification
$RSD$	Relative standard deviation



$s$	Standard deviation
SB	Secondary Batch
$s_h$	Standard deviation of the results of the samples tested to assess the homogeneity of the Secondary Batch
$s_{MB}$	Standard deviation of the $n_{MB}$ results of the samples of the Master Batch tested for the certification of the Secondary Batch
$s_{SB}$	Standard deviation of the $n_{SB}$ results of the samples tested for the characterisation of the Secondary Batch
$u_{CRM}$	Combined standard uncertainty of $KV_{CRM}$
$U_{CRM}$	Expanded uncertainty ( $k = 2$ , confidence level 95 %) of $KV_{CRM}$
$u_{char}$	Standard uncertainty of the result of the characterisation tests
$u_{char,rel}$	Relative standard uncertainty of the result of the characterisation tests
$u_h$	Contribution to uncertainty from heterogeneity
$u_i$	Value of uncertainty from contribution i
$u_{MB}$	Standard uncertainty of $KV_{MB}$
$u_{MB,rel}$	Relative standard uncertainty of $KV_{MB}$
% m/m	Mass fraction
$\bar{X}_{MB}$	Mean $KV$ value of the $n_{MB}$ measurements on samples of the Master Batch tested when characterising the Secondary Batch
$\bar{X}_{SB}$	Mean $KV$ value of the $n_{SB}$ results of the samples tested for the characterisation of the Secondary Batch
$\Delta h$	difference between the height of the centre of gravity of the pendulum prior to release and at the end of the half-swing during which the test sample is broken
$\nu_{RM}$	Effective number of degrees of freedom associated with the uncertainty of the certified value

## 1 Introduction: the Charpy pendulum impact test

The Charpy pendulum impact test is designed to assess the resistance of a material to shock loading. The test, which consists of breaking a notched bar of the test material using a hammer rotating around a fixed horizontal axis, is schematically presented in Figure 1.

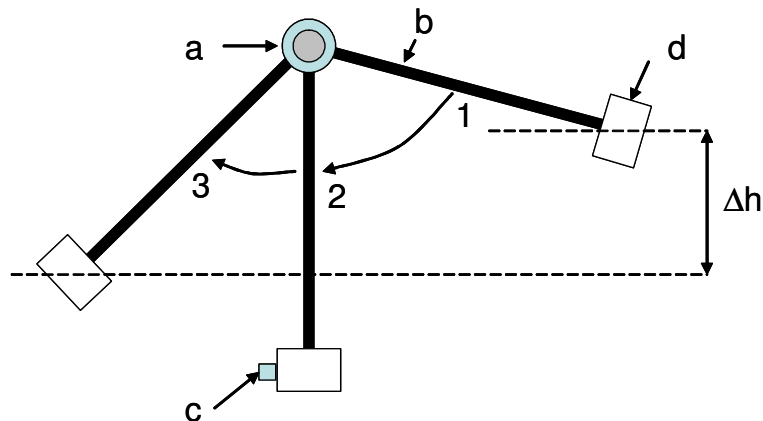


Figure 1: Schematic presentation of the Charpy pendulum impact test, showing a: the horizontal rotation axis of the pendulum, b: the stiff shaft on to which is fixed d: the hammer. The hammer is released from a well-defined height (position 1). When the hammer has reached maximum kinetic energy (shaft in vertical position 2), the hammer strikes c: the test sample, which is positioned on a support and against the pendulum anvils (not shown). The height reached by the hammer after having broken the sample (position 3) is recorded. The difference in height between position 1 and 3 ( $\Delta h$ ) corresponds with a difference in potential energy, and is a measure of the energy required to break the test sample.

The energy absorbed by the test sample is very dependent on the impact pendulum construction and its dynamic behaviour. Methods to verify the performance of an impact pendulum require the use of reference test pieces as described in European, ISO and other international standards [1, 2, 5]. The reference test pieces dealt with in this report comply with a V-notched test piece shape of well-defined geometry [1, 2], schematically shown in Figure 2.

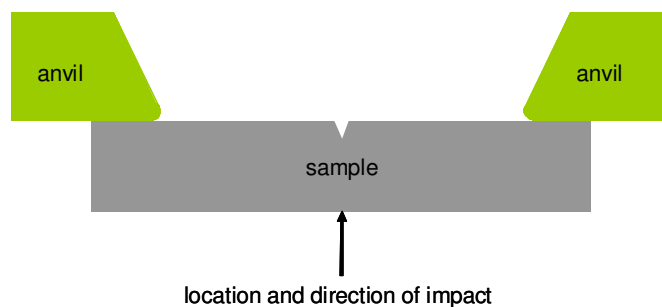


Figure 2: Schematic drawing of a V-notched Charpy sample (top-view), indicating the place and direction of impact.

## 2 The certification concept of Master Batch and Secondary Batch

### 2.1 Difference between Master and Secondary Batches

The BCR reports by Marchandise et al. [6] and Varma [7] provide details of the certification of BCR “Master Batches” (MB) of Charpy V-notch certified reference test pieces. The certified value of a Master Batch is obtained using an international laboratory intercomparison. Details of the most recent production of a Charpy Master Batch are given in [9].

This report describes the production of a “Secondary Batch” (SB) of Charpy V-notch certified reference test pieces at the Institute for Reference Materials and Measurements (IRMM) of the European Commission's (EC) Joint Research Centre (JRC). The work was performed in accordance with procedures described in the BCR reports [6] and [7]. The certification of a SB is based on the comparison of a set of SB test pieces with a set of test pieces from the corresponding MB under repeatability conditions on a single pendulum.

The BCR reports [6] and [7] were published in 1991 and 1999, respectively. Since 2000, the calculation of the certified value and the estimation of its uncertainty have been updated to an approach compliant with the ISO Guide to the Expression of Uncertainty in Measurement [8]. This revised approach was developed and presented by Ingelbrecht et al. [10, 11], and is summarised below.

### 2.2 Certification of a Secondary Batch of Charpy V-notch test pieces

The certified absorbed energy of a SB of Charpy V-notch reference test pieces ( $KV_{CRM}$ ) is calculated from the mean  $KV$ -value of a set of SB-samples ( $\bar{X}_{SB}$ ) tested on a single pendulum. This value  $\bar{X}_{SB}$  has to be corrected for the bias of this particular pendulum. The bias of the pendulum at the moment of testing the samples of the SB, is estimated by comparing the mean  $KV$ -value of a number of samples of the MB ( $\bar{X}_{MB}$ ), tested together with the SB samples under repeatability conditions, with the certified value of the MB ( $KV_{MB}$ ).  $KV_{CRM}$  is then calculated as follows [10]:

$$KV_{CRM} = \left[ \frac{KV_{MB}}{\bar{X}_{MB}} \cdot \bar{X}_{SB} \right] \quad \text{Eq. 1}$$

For this approach to be reliable, the pendulum used for the tests on MB and SB in repeatability conditions, must be well performing. In other words, the ratio  $\frac{KV_{MB}}{\bar{X}_{MB}}$  must be close to 1. IRMM now allows a difference of 5 % ( $KV_{MB} > 40$  J) or 2 J ( $KV_{MB} < 40$  J) between  $KV_{MB}$  and  $\bar{X}_{MB}$ , corresponding with the level of bias allowed for reference pendulums specified in EN 10045-2 [1] and ISO 148-3 [12].

Also, for reasons of commutability, a comparable response of the pendulum to the MB and SB samples is required. This is the reason why MB and SB samples are made from nominally the same steel. Moreover, it is checked that the ratio  $\frac{KV_{CRM}}{KV_{MB}}$  is close to 1. IRMM now allows a difference of 20 % ( $KV_{MB} > 40$  J) or 8 J ( $KV_{MB} < 40$  J)

between  $KV_{CRM}$  and  $KV_{MB}$  to ensure that the MB and SB samples have a comparable interaction with the pendulum.

### **2.3 Uncertainty of the certified value of a Secondary Batch of Charpy V-notch test pieces**

The uncertainty of the certified value of the SB is a combination of the uncertainties of the right-hand side factors in Eq. 1. It is clear that the MB-SB approach necessarily results in a larger uncertainty of the certified value of SB in comparison with the MB. The additional uncertainty depends on the uncertainty of the ratio  $\bar{X}_{MB} / \bar{X}_{SB}$ . The full measurement uncertainty of the values  $\bar{X}_{MB}$  and  $\bar{X}_{SB}$  is relatively large. However, when all conditions mentioned above (repeatability conditions, pendulum performance, and commutability between Secondary and Master Batch) are fulfilled, then the uncertainties of the values  $\bar{X}_{MB}$  and  $\bar{X}_{SB}$  have several contributions in common, in particular the uncertainty due to the bias of the pendulum. These shared uncertainty components do not contribute to the uncertainty of the ratio  $\bar{X}_{MB} / \bar{X}_{SB}$ , and only the standard deviations of the SB and MB results in the MB-SB comparison test need to be taken into account (see also Section 5.3). Thus, the MB-SB comparison approach can produce a value for the uncertainty of  $KV_{CRM}$  that is sufficiently small to meet the requirements of the intended use of the certified reference material (CRM).

## **3 Participants**

The processing of the SB (ERM<sup>®</sup>-FA013bf) test pieces was carried out by the Laboratoire National de Métrologie et d'Essais (LNE), Trappes (FR), using steel bars produced at Aubert&Duval, Ancizes and Gennevilliers (FR). The MB (ERM<sup>®</sup>-FA013ba) samples used in the characterisation of the SB were provided by IRMM, Geel (BE). The homogeneity of the SB was evaluated based on data obtained at LNE using a pendulum verified according to the criteria imposed by EN 10045-2 [1] and ISO 148-2 [2]. Characterisation of the SB was carried out at IRMM using a pendulum verified according to the criteria imposed by EN 10045-2 [1] and ISO 148-2 [2]. Data evaluation was performed at IRMM.

## **4 Processing**

The ERM<sup>®</sup>-FA013bf test pieces were prepared from AISI 4340 steel. The steel was cast and rolled into bars at Aubert&Duval (see section 4.1). Production of the test pieces from these bars was performed under the supervision of LNE (see sections 4.2, 4.3, 4.4 and 4.5).

### **4.1 Processing of hot-rolled bars**

The base material consisted of AISI 4340 steel, produced at Aubert&Duval. To limit the amount of impurities potentially affecting the homogeneity of the fracture resistance, the following compositional tolerances were imposed on the selected steel batch: Mn 0.7 – 0.8, Mo 0.23 - 0.28, Ni 1.7 – 1.85, P < 0.01, Si 0.2 – 0.35, S < 0.008 (in wt %), which is stricter than generally allowed for AISI 4340. The ingot was hot rolled, resulting in bars that were 6.4 m long and with a squared cross-section of 12 mm. For the ERM<sup>®</sup>-FA013bf batch, steel was used from ingot number SC1106W.

## **4.2 Heat treatment of hot-rolled bars**

The heat treatment of the hot-rolled bars was performed at Aubert&Duval. 15 bars were heat-treated together. Bars were placed onto rollers which slowly move the bars back and forth inside the furnace during the heat treatment to increase the homogeneity of the resulting microstructure. The first heat treatment was an austenisation treatment performed in a furnace of 'class 10 °C' <sup>1</sup> at 850 °C for 30 min. From this furnace, the bars were quenched into oil at 40 °C. After the oil-quench, the samples were annealed in a furnace of 'class 5 °C' at 350 °C for 120 min. After this annealing treatment, the samples were cooled down in air.

After heat treatment, a limited number of samples (5) were machined for a preliminary check of the obtained energy level. Results indicated an average *KV*-level (26.2 J) within the desired energy range (22.5 J to 27.5 J).

## **4.3 Machining of Charpy test pieces**

After the heat treatment the samples were machined to dimensional tolerances imposed in EN 10045-2 [1] and ISO 148-3 [12]. The batch code (BF30) and the individual sample code were engraved twice on one long face of each sample, on both sides of the notch, for fully traceable sample coding also after fracture ('30' indicates the nominal absorbed energy level (30 J); 'BF' is the letter code as assigned consecutively to batches of the same nominal absorbed energy). The V-notch was etched using an electro-erosion tool.

## **4.4 Quality control**

When all samples from the batch were fully machined, a selection of 25 samples was made. The dimensions of the 25 samples were checked on February 11, 2010 against the criteria specified in ISO 148-2 [3] (length  $55.0^{0.0}_{0.25}$  mm, height  $10.00 \pm 0.06$  mm, width  $10.00 \pm 0.07$  mm, notch angle  $45 \pm 1$  °, height remaining at notch root  $8.00 \pm 0.06$  mm, radius at notch root  $0.25 \pm 0.025$  mm, distance between the plane of symmetry of the notch and the longitudinal axis of the test piece  $27.5 \pm 0.2$  mm). None of the samples were outside the ranges specified in ISO 148-3 [12].

The 25 samples checked for geometrical compliance were impact tested on February 12, 2010 on the Tinius Olsen 358 Joules pendulum - which is one of the French reference pendulums - at LNE. The results are reported in certificate LNE n° G080477/DE/4 [13]. The average *KV* of the 25 samples was 28.47 J, which is sufficiently close to the desired energy range (22.5 J to 27.5 J). The standard deviation of the test results ( $s = 0.88$  J,  $RSD = 3.1$  %) was smaller than the maximum allowed 4 %. The variation was checked again during the certification tests at IRMM (see section 5).

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<sup>1</sup> In a furnace of 'class x °C', the variation of the temperature is smaller than x °C. The furnaces used have 10 heating zones. Each zone has 3 controlling thermocouples and 3 measurement thermocouples. These are regularly calibrated. When one faulty thermocouple is detected, it is replaced by a thermocouple produced with wire from the same roll. When a roll is exhausted, all thermocouples are replaced with new ones.

#### **4.5 Packaging and storage**

Finally, the samples were cleaned and packed in sets of 5, in oil-filled and closed plastic bags. These oil-filled bags, together with a label, again were packed in a sealed plastic bag, and shipped to IRMM. After arrival (February 2010), the 1390 samples (or 278 sets) were registered and stored at room temperature, pending distribution.

## **5 Characterisation**

### **5.1 Characterisation tests**

30 samples from ERM<sup>®</sup>-FA013bf (sets 1, 85, 148, 202, 236 and 269) were tested under repeatability conditions with 25 samples from MB ERM<sup>®</sup>-FA013ba (sets 7, 94, 165, 227 and 251), using the Instron Wolpert PW 30 (serial number 7300 H1527) machine of IRMM, an impact pendulum yearly verified according to procedures described in EN 10045-2 [1] and ISO 148-2 [2]. Tests were performed on March 24, 2010 (laboratory temperature  $20 \pm 1$  °C), in accordance with EN 10045-1 [3] and ISO 148-1 [4]. The measured absorbed energy values were corrected for friction and windage losses.

1 of the 30 samples tested for ERM<sup>®</sup>-FA013bf and 2 of the 25 samples tested for ERM<sup>®</sup>-FA013ba gave excessively high absorbed energy values. Inspection of the broken samples reveals the same type of damage as observed on previous FA013 batches. After testing, all Charpy samples show 'first-strike' marks: these are the marks caused by the interaction between sample and anvils during the first and intended hammer impact. Upon fracture, the broken half samples loose contact with hammer and anvils and follow one of a variety of possible trajectories, away from the pendulum, depending on the properties of both pendulum and test material. Some samples show 'second-strike' marks. These are marks caused by a second impact of the already broken half samples back onto the anvils. This phenomenon has been described by Schmieder et al. [14]. Most of the broken FA013ba and FA013bf samples show second-strike marks. A mere second impact of the broken half samples onto the anvils does not affect the measured KV value, since it does not involve or slow down the swinging pendulum.

For 3 samples, a significant post-fracture deformation mark on one of the long edges of the notched face of the sample was observed. Systematically, this indent is accompanied by a broader deformation of the diagonally opposite long edge (on the face of the sample opposite the notch). This suggests that the samples, during the second strike onto the anvils, were briefly caught between anvil and a part of the swinging pendulum. This induces erroneously high measured absorbed energy values. For this technical reason, 3 data were eliminated from the analysis. All other data were accepted. An analysis of the normality of the distribution of remaining KV values did not indicate a skewed distribution towards higher or lower absorbed energy values.

The same parasitic energy absorbing phenomenon occurred also for some but not all of the other batches which were prepared previously. Also, the phenomenon did not occur during the pre-acceptance tests at LNE. Furthermore, the FA013ba batch was tested on 12 pendulums during a laboratory intercomparison, revealing that post-fracture energy absorption occurs only for a minority of the pendulums involved (2 out of 12).

The above observations indicate that excessive post-fracture energy absorption affecting the measured KV values can occur, but only for a limited number of combinations of pendulum and material. In each case, excessively high measured values are easily related to large post-fracture deformation marks on the broken samples. Users of ERM-FA013bf reference materials encountering outlier values that can be related to post-fracture indentation marks on the broken samples are requested to eliminate the corresponding data from the analysis of their results.

The comparison of the indirect verification results with the certified value and uncertainty is based on the mean of the 5 measured KV values, because this is the sample size for which the uncertainty of the certified value has been calculated. If outlier values have to be eliminated because of post-fracture indentation, the corresponding expanded uncertainty for  $n = 4$  remaining results is  $U = 1.2$  J, for  $n = 3$  remaining results  $U = 1.3$  J.

The accepted data obtained on individual test pieces are shown in Figure 3 and in Annex 1. In Figure 3 linear trend-lines are added to illustrate the absence of a significant trend in the results with the analysis sequence. The results of the measurements are summarised in Table1.

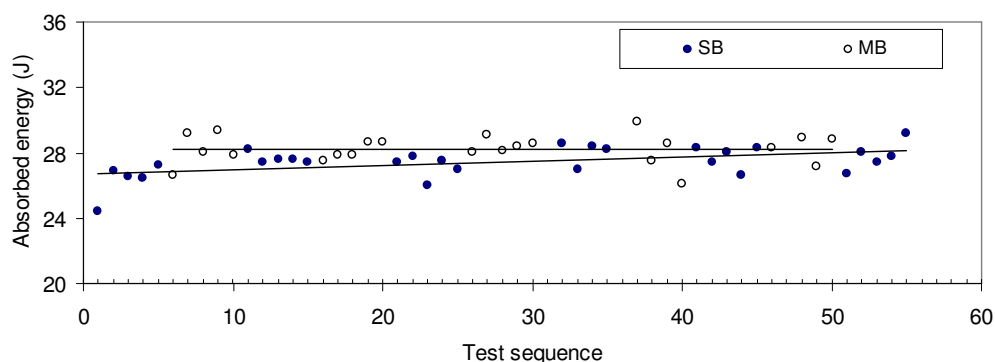


Figure 3: Absorbed energy values of the 29 test pieces of ERM<sup>®</sup>-FA013bf and 23 test pieces of ERM<sup>®</sup>-FA013ba displayed in the actual test sequence.

Table 1: Results of characterisation of Batch ERM<sup>®</sup>-FA013bf by comparison with Master Batch ERM<sup>®</sup>-FA013ba.

	Number of test pieces	Mean value	Standard deviation	Relative standard deviation
	$n_{MB}$ or $n_{SB}$	$\bar{X}_{MB}$ or $\bar{X}_{SB}$ (J)	$s_{MB}$ or $s_{SB}$ (J)	$RSD$ (%)
<b>ERM<sup>®</sup>-FA013ba (MB)</b>	23	28.23	0.88	3.1
<b>ERM<sup>®</sup>-FA013bf (SB)</b>	29	27.43	0.93	3.4

The SB-results meet the EN 10045-2 and ISO 148-3 acceptance criteria for a batch of reference materials ( $s_{SB} < 2$  J), as well as the more stringent acceptance criterion ( $s_{SB} < 1.5$  J) contractually fixed between IRMM and its sample supplier for batches of this nominal absorbed energy level (30 J).

## 5.2 Data from Master Batch ERM<sup>®</sup>-FA013ba

To calculate  $KV_{CRM}$  for ERM<sup>®</sup>-FA013ba one needs  $KV_{MB}$  of the MB used, i.e. ERM<sup>®</sup>-FA013ba. Table 2 shows the main MB-data, taken from the Certificate of Analysis of ERM<sup>®</sup>-FA013ba (Annex 2).

Table 2: Data from the certification of Master Batch ERM<sup>®</sup>-FA013ba.

	<b>Certified absorbed energy of Master Batch</b>	<b>Standard uncertainty of <math>KV_{MB}</math></b>	<b>Standard uncertainty of <math>KV_{MB}</math></b>
	$KV_{MB}$ (J)	$u_{MB}$ (J)	$u_{MB,rel}$ (%)
<b>ERM<sup>®</sup>-FA013ba</b>	28.46	0.2	0.8

## 5.3 Calculation of $KV_{CRM}$ and of $u_{char}$

From the data in Table 1 and Table 2, and using Eq. 1, one readily obtains that  $KV_{CRM} = 27.7$  J. The uncertainty associated with the characterisation of the SB,  $u_{char}$ , is assessed as in Eq. 2 [11], which sums the relative uncertainties of the three factors in Eq. 1:

$$u_{char} = KV_{CRM} \sqrt{\frac{u_{MB}^2}{KV_{MB}^2} + \frac{s_{SB}^2}{n_{SB} \cdot \bar{X}_{SB}^2} + \frac{s_{MB}^2}{n_{MB} \cdot \bar{X}_{MB}^2}} \quad \text{Eq. 2}$$

$\bar{X}_{SB}$  and  $\bar{X}_{MB}$  were obtained under repeatability conditions. Therefore, the uncertainty of the ratio  $\bar{X}_{SB} / \bar{X}_{MB}$  is not affected by the contributions from reproducibility and bias of the pendulum used to compare MB and SB. Table 3 summarises the input quantities of the  $u_{char}$  uncertainty budget, their respective statistical properties, and shows how they were combined. The effective number of degrees of freedom for  $u_{char}$  is obtained using the Welch-Satterthwaite equation [8].



Table 3: Uncertainty budget for  $u_{char}$

symbol	source of uncertainty	measured value (J)	standard uncertainty (J)	probability distribution	divisor	sensitivity coefficient	relative uncertainty (%)	degrees of freedom
$KV_{MB}$	Certification of MB	28.46	0.23	normal	1	1	0.81	14
$\bar{X}_{SB}$	comparison of SB and MB in repeatability conditions	27.43	0.17	normal	1	1	0.63	28
$\bar{X}_{MB}$		28.23	0.18	normal	1	1	0.65	22
$u_{char,rel}$ (%)							1.21	48
$u_{char}$ (J)							0.33	

## 6 Homogeneity

The test pieces are sampled from the SB, which is sufficiently, but not perfectly, homogeneous. Therefore, an appropriate homogeneity contribution  $u_h$  to the uncertainty of the certified value is required.  $u_h$  is related to  $s_h$ , the standard deviation between the samples in the SB (*sample-to-sample heterogeneity*), but also depends on the number of samples over which the  $KV$ -value is averaged. EN 10045-2 [1] and ISO 148-2 [2] specify that the pendulum verification must be performed using 5 test pieces, which is why a CRM-unit consists of a set of 5 test pieces. The appropriate uncertainty contribution must be an estimate of the *set-to-set heterogeneity*, which in

the case of a set of 5 test pieces can be calculated as  $u_h = \frac{s_h}{\sqrt{5}}$ .

Here,  $u_h$  is estimated from the results obtained at LNE on February 12, 2010 ( $s_h = 0.88$  J), which leads to  $u_h = \frac{s_h}{\sqrt{5}} = 0.40$  J. As is required for a homogeneity test, the

samples were randomly selected from the whole batch. The number of samples tested (25) is sufficiently large to reflect the homogeneity of the full SB (1390 samples). It can be noted that  $u_h$  is probably a slight overestimation, since it contains also the repeatability of the instrument. However, the latter cannot be separated or separately measured.

## 7 Stability

The stability of the absorbed energy of Charpy V-notch certified reference test pieces was first systematically investigated for samples of nominally 120 J by Pauwels et al., who did not observe measurable changes of absorbed energy [15]. New evidence for the stability of the reference test pieces produced from AISI 4340 steel of lower energy levels (nominally 15 J, 30 J and 100 J) has been obtained recently, during the International Master Batch (IMB) project [16]. In the IMB-project, the stability of the certified test pieces is judged from the change of the mean of means of the absorbed energy obtained on 7 reference pendulums over a three year period. None of the three regression slopes for the tested energy levels was statistically significant at the 0.05 level. Given the large sample-to-sample heterogeneity and the limited number of samples (5) in a CRM unit, the uncertainty contribution from instability is considered to be insignificant in comparison to that of homogeneity.

The main reason for the microstructural stability of the certified reference test pieces is the annealing treatment to which the samples were subjected after the austenisation treatment. Annealing is performed at temperatures where the equilibrium phases are the same as the (meta-)stable phases at ambient temperature ( $\alpha$ -Fe and  $\text{Fe}_3\text{C}$ ). The only driving force for instability stems from the difference in solubility of interstitial elements in the  $\alpha$ -Fe matrix, between annealing and ambient temperature. Relaxation of residual (micro-)stress by short-range diffusion or the additional formation or growth of precipitates during the shelf-life of the certified reference test pieces is expected to proceed but slowly.

Rather than neglecting the stability issue, efforts are spent to better establish the stability of the certified values of batches of Charpy CRMs. Until such further notice, it is decided to specify a limited shelf-life. A period of 10 years is chosen, counting from the date of the characterisation tests on the SB. Since batch ERM<sup>®</sup>-FA013bf was characterised in March, 2010, the validity of the certificate reaches until March, 2020.

## 8 Calculation of certified value, combined and expanded uncertainty

As shown in 5.2,  $KV_{\text{CRM}} = 27.7$  J. The uncertainty of the certified value is obtained by combining the contributions from the characterisation study,  $u_{\text{char}}$ , and from the homogeneity assessment,  $u_{\text{h}}$ , as is summarized in the following uncertainty budget (Table 4).

The relevant number of degrees of freedom calculated using the Welch-Satterthwaite equation [8], is sufficiently large ( $\nu_{\text{RM}} = 56$ ) to justify the use of a coverage factor  $k = 2$  to expand the confidence level to about 95 %. The obtained expanded uncertainty provides justification for the SB-MB approach followed:  $U_{\text{CRM}}$  is sufficiently smaller than the verification criterion of 4 J or even 2 J for reference pendulums.

Table 4: Uncertainty budget of  $KV_{CRM}$

symbol	source of uncertainty	absolute value (J)	$u_i$ (J)	degrees of freedom
$u_{char}$	characterisation of SB	0.33	0.33	48
$u_h$	homogeneity of SB	0.40	0.40	24
Combined standard uncertainty, $u_{CRM}$			0.52 J	56
Expanded Uncertainty, $k = 2$ , $U_{CRM}$			1.1 J	$\nu_{RM}$

## 9 Traceability

The certified property is defined by the Charpy pendulum impact test procedure described in EN 10045-1 [3] and ISO 148-1 [4].

The certified value of the MB ERM<sup>®</sup>-FA013ba is traceable to the SI, since it was obtained using an interlaboratory comparison, involving a representative selection of qualified laboratories performing the tests in accordance with the standard procedures and using instruments verified and calibrated with SI-traceable calibration tools.

The certified value of ERM<sup>®</sup>-FA013bf is made traceable to the SI-traceable certified value of the MB by testing SB and MB samples in repeatability conditions on an impact pendulum verified and calibrated with SI-traceably calibrated tools. Therefore, the certified value of ERM<sup>®</sup>-FA013bf is traceable to the International System of Units (SI) via the corresponding Master Batch ERM<sup>®</sup>-FA013ba of the same nominal absorbed energy (30 J). Absorbed energy  $KV$  is a method-specific value, and can only be obtained by following the procedures specified in EN 10045-1 [3] and ISO 148 [4].

## 10 Commutability

The intended use of the certified reference test pieces is the verification of Charpy impact pendulums. During the certification of the MB, different pendulums were used, each equipped with an ISO-type striker of 2 mm tip radius. Until further notice, the certified values are not to be used when the test pieces are broken with an ASTM-type striker of 8 mm tip radius, although the expected differences can be small for materials with relatively low absorbed energy such as the ERM<sup>®</sup>-FA013 batches of 30 J nominal absorbed energy [15].

## 11 Summary of results

The certified value and associated uncertainties are summarized in Table 5.

Table 5: Certified value and associated uncertainties for ERM<sup>®</sup>-FA013bf.

Steel Charpy V-notch test pieces		
	Certified value <sup>2)</sup> [J]	Uncertainty <sup>3)</sup> [J]
Absorbed energy (KV) <sup>1)</sup>	27.7	1.1
<p>1) The absorbed energy (KV) is a method defined measurand. KV is the impact energy required to break a V-notched bar of standardised dimensions, as defined in EN 10045-1 and ISO 148-1. The certified value is valid only for strikers with a 2 mm tip radius, and in the temperature range of (20 ± 2) °C.</p> <p>2) The certified value of ERM<sup>®</sup>-FA013bf, and its uncertainty, are traceable to the International System of Units (SI), via the master batch ERM<sup>®</sup>-FA013ba of the same nominal absorbed energy (30 J) by testing samples of ERM<sup>®</sup>-FA013ba and ERM<sup>®</sup>-FA013bf under repeatability conditions on an impact pendulum verified and calibrated with SI-traceably calibrated tools.</p> <p>3) Estimated expanded uncertainty of the mean KV of the 5 specimens (delivered as 1 set), with a coverage factor <math>k = 2</math>, corresponding to a level of confidence of about 95 %, as defined in the ISO/IEC Guide 98-3:2008; Uncertainty of measurement – Part 3: Guide to the expression of uncertainty in measurement (GUM). The number of degrees of freedom of the certified uncertainty, <math>\nu_{RM} = 56</math>.</p>		

## 12 Instructions for use

### 12.1 Intended use

Samples of ERM<sup>®</sup>-FA013bf correspond to the ‘(certified) BCR test pieces’ as referred to in EN 10045-2 [1], as well as to the ‘certified reference test pieces’ as defined in ISO 148-3 [12]. Sets of five of these certified reference test pieces are intended for the indirect verification of impact testing machines with a striker of 2 mm tip radius according to procedures described in detail in EN 10045-2 [1] and ISO 148-2 [2].

The indirect verification provides an assessment of the bias of the user’s Charpy pendulum impact machine. This bias assessment can be used in the calculation of the measurement uncertainty of Charpy tests on the pendulum after indirect verification. Such uncertainty calculation requires the certified value, the associated uncertainty, and in some cases also the degrees of freedom of the uncertainty, all given on page 1 of the certificate.

### 12.2 Sample preparation

Special attention is drawn to cleaning of the specimens prior to the tests. It is mandatory to remove the oil from the sample surface prior to testing, without damaging the edges of the sample. Between the moment of removing the protective oil layer and the actual test, corrosion can occur. This must be avoided by limiting this period of time, while keeping the sample clean.

The following procedure is considered a good practice.

1. First use absorbent cleaning-tissue to remove the excess oil. Pay particular attention to the notch of the sample, but do not use hard (e.g. steel) brushes to remove the oil from the notch.
2. Submerge the samples in ethanol for about 5 min. Use of ultrasonication is encouraged, but only if the edges of the samples are prevented from rubbing against each other. To reduce the consumption of solvent, it is allowed to make a first cleaning step with detergent, immediately prior to the solvent step.
3. Once samples are removed from the solvent, only manipulate the samples wearing clean gloves. This is to prevent development of corrosion between the time of cleaning and the actual test.
4. Before testing, bring the specimens to the test temperature ( $20 \pm 2$ ) °C. To assure thermal equilibrium is reached, move the specimens to the test laboratory at least 3 h before the tests.

### **12.3 Pendulum impact tests**

After cleaning, the 5 samples constituting a CRM-unit need to be broken with a pendulum impact test machine in accordance with EN 10045-2 [1] or ISO 148-2 [2] standards. Prior to the tests, the anvils must be cleaned. It must be noted that Charpy test pieces sometimes leave debris on the Charpy pendulum anvils. Therefore, the anvils must be checked regularly and if debris is found, it must be removed. The uncertainty of the certified value applies to the mean of the 5 KV-values.

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## Annex 1

Results of characterisation measurements of ERM®-FA013bf as measured according to EN 10045-1 and ISO 148 at IRMM, March 24, 2010.

	<b>Master Batch ERM®-FA013ba</b>	<b>Secondary Batch ERM®-FA013bf</b>
	<b><i>KV (J)</i></b>	<b><i>KV (J)</i></b>
1	26.61	24.38
2	29.21	26.92
3	28.03	26.53
4	29.34	26.47
5	27.90	27.25
6	27.51	28.22
7	27.90	27.39
8	27.90	27.64
9	28.69	27.64
10	28.69	27.39
11	28.03	27.39
12	29.08	27.77
13	28.16	25.98
14	28.42	27.51
15	28.55	27.00
16	29.87	28.55
17	27.51	26.99
18	28.55	28.35
19	26.10	28.22
20	28.29	28.29
21	28.95	27.39
22	27.13	28.03
23	28.82	26.61
24		28.29
25		26.74
26		28.03
27		27.39
28		27.77
29		29.21
30		
<b>Mean (J)</b>	<b>28.23</b>	<b>27.43</b>
<b>Standard deviation (J)</b>	<b>0.88</b>	<b>0.93</b>
<b><i>RSD (%)</i></b>	<b>3.1</b>	<b>3.4</b>



## Annex 2



# CERTIFICATE OF ANALYSIS

ERM®- FA013ba

STEEL		
	Impact toughness	
	Certified value <sup>2)</sup> [J]	Uncertainty <sup>3)</sup> [J]
Absorbed energy (KV) <sup>1)</sup>	28.46	0.23
<p>1) The absorbed energy (KV) is procedurally defined and refers to the impact energy required to break a V-notched bar of standardised dimensions, as defined in EN 10045-1 and ISO 148-1.</p> <p>2) The certified value is estimated as the mean of means of absorbed energies measured at 15 laboratories. At each laboratory, 20 test pieces were broken. The instruments used by these laboratories are regularly verified with equipment that is calibrated in a manner that is traceable to the International System of Units (SI). Therefore, the certified value is traceable to the International System of Units (SI).</p> <p>3) Standard uncertainty <math>u</math> of the certified mean absorbed energy of batch ERM-FA013ba, estimated as the standard deviation of the mean of the 15 laboratory mean values, corresponding with a confidence level of about 68 %.</p>		

This certificate is valid until January 2018.

### NOTE

European Reference Material ERM®-FA013ba was produced and certified under the responsibility of the Institute for Reference Materials and Measurements of the European Commission's Joint Research Centre according to the principles laid down in the technical guidelines of the European Reference Materials® co-operation agreement between BAM-IRMM-LGC. Information on these guidelines is available on the internet (<http://www.erm-crm.org>).

Accepted as an ERM®, Geel, January 2009.

Signed: \_\_\_\_\_

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Registration No. 268-TEST  
ISO Guide 34 for the  
production of reference materials

All following pages are an integral part of the certificate.

Page 1 of 3

## DESCRIPTION OF THE SAMPLE

A unit consists of five Charpy V-notch test pieces, which are rectangular steel bars of nominal dimensions 55 mm x 10 mm x 10 mm, with one V-notch, accurately machined to tolerances imposed in EN 10045-2 and ISO 148-3. The five specimens are packed together in a plastic bag filled with oil to prevent oxidation.

## ANALYTICAL METHOD USED FOR CERTIFICATION

Charpy pendulum impact tests in accordance with EN 10045-1 and ISO 148-1, using pendulum impact machines with a 2 mm striker tip radius.

## PARTICIPANTS

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- Universität Stuttgart, Materialprüfungsanstalt, Stuttgart (DE)\* (DAP-PL-2907.02)
- VTT, Espoo (FI)

\* Measurements within the scope of accreditation to ISO 17025.

## SAFETY INFORMATION

Precautions need to be taken to avoid injury of the operator by broken specimens when operating the Charpy impact pendulum.

## INSTRUCTIONS FOR USE

Samples of ERM-FA013ba correspond with the '(certified) BCR test pieces' as referred to in EN 10045-2 (Method for the verification of impact testing machines), as well as with the 'certified reference test pieces' as defined in ISO 148-3 (Preparation and characterisation of Charpy V reference test pieces for verification of test machines).

The ERM-FA013ba batch is one of the 'Master Batches'. Master Batch test pieces are not for sale. They are intended solely to traceably certify Secondary Batches of the same nominal absorbed energy (here 30 J). The certified value and its associated uncertainty of the Master Batch are used in the calculation of the certified value and combined and expanded uncertainty of a set of 5 specimens from a Secondary Batch.

When characterising a secondary batch, a number of Master Batch test pieces are broken under repeatability conditions together with a selection of samples from the secondary batch. Special attention is drawn to the cleaning and conditioning of the specimens prior to testing. It is mandatory to remove the oil from the sample surface prior to testing, without damaging the edges of the sample. Between the moment of

removing the protective oil layer and the actual test, corrosion can occur. This must be avoided by limiting this period of time, while keeping the sample clean.

The following cleaning and conditioning procedure is considered to be good practice.

1. First use absorbent cleaning-tissue to remove the excess oil. Pay particular attention to the notch of the sample, but do not use hard (e.g. steel) brushes to remove the oil from the notch.
2. Submerge the samples in technically pure ethanol for about 5 minutes. Use of ultrasonication is encouraged, but only if the edges of the samples are prevented from rubbing against each other. To reduce the consumption of solvent, it is allowed to make a first cleaning step with detergent, immediately prior to the solvent step.
3. Once samples are removed from the solvent, only manipulate the samples wearing clean gloves. This is to prevent development of corrosion between the time of cleaning and the actual test.
4. Before testing, bring the specimens to the test temperature ( $20 \pm 2$  °C). To assure thermal equilibrium is reached, move the specimens to the test laboratory at least 3 h before the tests.

After cleaning and equilibration, the samples need to be broken with a pendulum impact test machine operated in accordance with EN 10045-1 or ISO 148-1 standards. Prior to the tests, the anvils must be cleaned. It must be noted that Charpy test pieces sometimes leave debris on the Charpy pendulum anvils. Therefore, the anvils must be checked regularly and if debris is found, it must be removed.

For some pendulums and for some samples, post-fracture interaction between broken samples and pendulum hammer can affect the measured KV values. The resulting excessively high values can be related to indentations and deformations of the broken samples. Outlier values that can be related to post-fracture indentation marks on the broken samples must be eliminated from the analysis of the results.

## STORAGE

Specimens should be kept at room temperature in their original packing until used. However, the European Commission cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially of opened samples.

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## NOTE

A detailed technical report can be obtained from the Joint Research Centre, Institute for Reference Materials and Measurements on request.

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**Abstract**

This certification report describes the processing and characterisation of ERM<sup>®</sup>-FA013bf, a batch of Charpy V-notch certified reference test pieces. Sets of five of these test pieces are used for the verification of pendulum impact test machines according to EN 10045-2 (Charpy impact test on metallic materials, Part 2. Method for the verification of impact testing machines [1]) or according to ISO 148-2 (Metallic materials - Charpy pendulum impact test – Part 2: Verification of test machines [2]).

The certified value for *KV* (= energy required to break a V-notched test piece using a pendulum impact test machine) is 27.7 J. The associated uncertainty (1.1 J,  $k = 2$  corresponding to a confidence level of about 95 %) is calculated for the mean of a set of five test pieces.

The absorbed energy (*KV*) is procedurally defined and refers to the impact energy required to break a V-notched bar of standardised dimensions, as defined in EN 10045-1 [3] and ISO 148-1 [4]. The certified value of ERM<sup>®</sup>-FA013bf is traceable to the SI, via the SI-traceable certified value of the master batch ERM<sup>®</sup>-FA013ba, by testing samples of ERM<sup>®</sup>-FA013ba and ERM<sup>®</sup>-FA013bf under repeatability conditions on an impact pendulum verified and calibrated with SI-traceably calibrated tools. The certified value is valid only for strikers with a 2 mm tip radius. The certified value is valid at  $(20 \pm 2)$  °C.

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